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(54) **Generator of coherent radiation using an intercalation compound.**

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WO-A-86/01046
FR-A- 2 532 929

CHEMICAL ABSTRACTS, vol. 104, no. 16, 21st April 1986, page 731, abstract no. 140869g, Columbus, Ohio, US; S. Tanuma et al.: "Synthesis and electronic properties of tetrachlorosilane-GIC"

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CHEMICAL ABSTRACTS, vol. 99, no. 20, 14th November 1983, page 643, abstract no. 168329g Columbus, Ohio, US; V.L. Solozhenko et al.: "Introduction of tin (IV) chloride into graphite"	
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Description

BACKGROUND OF THE INVENTION

This invention relates to a generator of coherent radiation using intercalation compounds, more particularly intercalation compounds obtained by intercalating halides of elements belonging to the group IVa of the periodic table or sodium in layer crystal substances. Such compounds have excellent properties in crystal characteristics, electric conductivity, electric dielectricity, heat conductivity, etc., and a solid state coherent radiation equipment using such an intercalation compound is particularly suitable for emitting coherent X-rays.

Heretofore, as layer crystal substances, there have been known diamond and graphite consisting of carbon atoms. On the other hand, layer crystal substances cannot be theoretically formed from Si, Ge and the like which are known as semiconductor substances. Thus, various studies have been made as to intercalation compounds obtained by intercalating other atoms or compounds among sheets of carbon atoms of graphite. But there has been no report as to the intercalation of the elements of the group IVa of the periodic table and halides thereof.

Chemical Abstracts, vol. 99, No. 20, page 643, No. 168329g refers to a layer structure comprising graphite intercalated with $SnCl_4$. X-rays and thermal analyses are used to determine properties of such a structure.

FR-A-2 532 929 discloses a further intercalate containing a sodium halide in the form of $C_{10}F(NaF)_{0.15}$ and is similarly concerned with the determination of such a structure by X-rays.

On the other hand, as generators of coherent radiation using a solid, semiconductor lasers have been known which emit radiation at wavelengths essentially from 750 nm to 1.5 μm.

Summary of the Invention

It is an object of the present invention to provide a generator of coherent radiation at shorter wavelengths than obtained by semiconductor lasers, particularly coherent X-rays having a wavelength of 1 nm or less.

This object is met by the device defined in claim 1.

BRIEF DESCRIPTION OF THE DRAWINGS

Figs. 1, 2, 3, 12 and 13 are schematic cross-sectional views showing apparatuses for producing the intercalation compounds.

Figs. 4 and 6 are schematic cross-sectional views showing the structures of intercalation compounds.

Figs. 5, 7 and 14 are schematic cross-sectional views showing devices for emitting coherent radiation by using the intercalation compounds.

Figs. 8 and 9 are schematic cross-sectional views showing optical resonators using an intercalation compound.

Figs. 10, and 11 are cross-sectional views showing intercalation compounds covered with a coating.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The intercalation compounds used in this invention comprises at least one atomic host layer of a layered crystalline substance and at least one molecular layer of a compound as an intercalant, both layers being positioned alternately.

As the layered crystalline substance, there can be used graphite, GaS, InS, InSe, HgI_2, and PbI_2.

As the intercalant, there can be used halides of elements belonging to the group IV of the periodic table, particularly the group IVa, i.e., Si, Ge, Sn, Pb, and halides of sodium. As the halides, there can be used chlorides, bromides, iodides, and fluorides. When Si, Ge, Sn, Pb, or Na alone is intercalated in graphite, it is not positioned at the centers of hexagons of carbon atoms constituting graphite, which results in strains in the crystal that destroy it. In contrast, when the elements belonging to the group IVa and Na are intercalated in the form of compounds of halides thereof, these compounds can be positioned at the centers of hexagons of carbon atoms to stabilize the resulting intercalation compound. Among these halides, $SiCl_4$, $GeCl_4$, $SnCl_4$, $PbCl_4$, NaBr, $GeBr_4$, NaCl are preferable.

The intercalation compound used in this invention has a structure, for example, as shown in Figs. 4(a) to 4(d), wherein 22 is at least one atomic layer, 23 is at least one molecular layer, 20 are atoms (e.g. carbon atom) forming a layered crystalline substance (e.g. graphite shown by a mark X) and 21 is an intercalated compound (shown by a mark o). As shown in Fig. 4, the number of atomic layers can be one as shown in Figs. 4(c) and 4(d) or three as shown in Figs. 4(a) and 4(b), but, needless to say, two or any other number may also be used. Further, the number of molecular layers can be one as shown in Figs. 4(a) and 4(c) or two as shown in Figs. 4(b) and 4(d), but needless to say, other numbers are possible.

The intercalation compound of this invention can be produced by placing the layered crystalline substance such as graphite in an atmosphere of vapor of the intercalant, i.e., a halide of Na or an element belonging to the group IV, or placing the layered crystalline substance in an atmosphere of

vapor of the intercalant and a halogen gas such as chlorine gas, bromine gas, etc. The latter process is particularly suited for extremely stably intercalating a halide of Na or an element belonging to the group IVa in graphite, particularly intercalating $SiCl_4$ using Cl_2 gas or intercalating NaBr using Br_2 gas.

In order to intercalate the halides of Na or a group IV element in graphite, the compound to be intercalated and graphite are placed in a reaction vessel as shown in Fig. 1, 2, 3, 12 or 13 and heated at a temperature of 100 to 500 °C, preferably 150 to 400 °C. After the intercalation, the reaction vessel is force - cooled, preferably to a supercooled state in order to prevent the release of the intercalated compound during the cooling. Further, when the cooling is carried out too rapidly, there is a danger of cracks caused in the crystal. Therefore, it is preferable to use a cooling rate faster than the heating rate (a temperature rise rate) employed above. For example, a cooling rate is preferably 0.1 to 10 °C/min to yield particularly stable intercalation compounds. In the case of intercalating $SiCl_4$, NaBr or $GeBr_4$, a cooling rate of 0.5 to 2 °C/min is particularly preferable.

In order to improve the stability of the intercalation compound used in this invention, it is possible to provide it with a covering. As the covering material, there can be used an inorganic material such as boron nitride (BN), silica (SiO_2), etc., and an organic resin such as a polyimide resin, etc. Such a covering can be formed directly on side faces perpendicular to the atom layer of the intercalation compound as shown in Fig. 10, or on front and back faces parallel to the atom layer of the intercalation compound as shown in Fig. 9. Further, as shown in Fig. 11, the whole surfaces of the intercalation compound can be covered. In the case shown in Fig. 10, since there is no covering at the faces parallel to the atom layer of the intercalation compound, the loss of excitation energy and radiation is small but there is a tendency to cause a pollution problem. In the case of Fig. 11, it is preferable to use a covering material low in absorption of excitation energy and radiation. Needless to say, the material covering the faces parallel to and that covering the side faces perpendicular to the atom layer of the intercalation compound may be different.

In the case of intercalating a halide of Si, when H_2O is present in the atmosphere for heating the layered crystalline compound such as graphite, a film of SiO_2 is formed on the surface of the resulting intercalation compound. Said SiO_2 film can be a covering for improving the stability.

The material and the thickness of the covering should be selected properly so as not to impair the effects of this invention due to excessive absorption of pumping energy.

The intercalation compounds used in this invention have excellent properties; for example, that obtained by intercalating $SiCl_4$ in graphite is large in photoconductivity and strong in anisotropy in the photoconductivity, that obtained by intercalating $GeCl_4$ is large in electric conductivity, that obtained by intercalating $SnCl_4$ or $PbCl_4$ is high in the critical temperature as to superconductivity.

A solid-state X-ray laser can be formed by applying a diffraction grating, and the like, by using these intercalation compounds.

In an X-ray laser, since the distance between the molecule layers takes a predetermined value in the above intercalation compound, this can be regarded as a periodic distributed feedback structure; this permits X-ray laser action having a wavelength corresponding to such a distance. Further, since the distance between the molecule layers can be changed depending on the conditions for producing samples, it is possible to realize X-ray laser beams having various wavelengths.

In addition, by using the above intercalant compounds, there is provided a novel laser as shown in Fig. 5. In Fig. 5, numeral 101 denotes the intercalation compound, numeral 102 a line perpendicular to the atom layer in the intercalation compound, numeral 103 a vector showing exciting energy, numeral 104 coherent radiation, and θ the angle between the perpendicular line 102 and the incident direction of exciting (or pumping) light 103.

The present inventors noted that the transmittance of light through a substance is proportional to the cube of the atomic (Z) number of the element constituting the substance and that a layer crystal substance consisting of an element having a small atomic weight such as graphite is transparent. When exciting energy is supplied to an intercalation compound having intercalated molecule layers of a compound containing an element having a larger atomic weight such as Si between the atom layers of graphite so as to excite the core electrons of the element constituting the intercalation compound, there takes place a resonance action for macroscopic fluorescence X-ray (0.66 nm) generated at the time of relaxation of the layers of the intercalation compound due to the slowness of the relaxation of a macroscopic dipole correlation of the layers forming the group of molecule layers compared with the case of monomolecule layer, and coherent electromagnetic waves are generated similar to the classical case of cavity resonance. When the layer crystal substance is graphite, the wavelength of the generated electromagnetic waves becomes 0.66 nm.

Further, when the pumping power becomes large, there takes place a population inversion of electrons to provide a super radiant X-ray laser.

This is a kind of travelling-wave type laser.

The intercalation compound used in such a laser has a structure as shown in Fig. 6, wherein numeral 105 denotes an monocrystal atom layer of a crystal substance such as graphite and three molecular layers 106 of intercalated compound are interposed therebetween. A single molecule layer of intercalation compound is easily intercalated between the atom layers, but a plurality of molecular layers can be intercalated between the atomic layers. Since the absorption coefficient of an atom for a beam of radiation having a certain wavelength is proportional to the cube of the atomic number (Z), a beam of radiation, particularly X-rays, is hardly absorbed in the carbon atom layer (C: Z = 6), which is, therefore, regarded as transparent. When the molecule layer consists of elements such as Si (Z = 14) or Ge (Z = 32) and Cl (Z = 17), electrons of these elements are excited, and the beam generated at the time of relaxation thereof resonates in a resonator constituted by the intercalation compound to generate a standing wave. When the pumping power becomes larger than the threshold, a super radiant is generated. Thus, a laser action is admitted in the intercalation compound.

Extremely stable optical resonators can be obtained with acceptor type intercalation compounds, which are obtained by using as the compound for forming the molecule layer halides of Na and elements belonging to the group IVa.

A laser action is also found when there is used an optical resonator as shown in Fig. 8, wherein numeral 101 is the intercalation compound and numeral 107 is a crystal layer of an element belonging to the group IVa such as Si, etc., or Na.

The resonance wavelength of the optical resonator can be obtained theoretically from lattice spaces and electron states of constituting substances such as graphite, the IV group elements, Na, element and halogen atoms, and the structure of the intercalation compound (the state of intercalation such as the distance between the two molecule layers). For example, the wavelength is 0.66 nm when $SiCl_4$ is intercalated every three layers of the atom layers of graphite, and 0.29 nm when NaBr is intercalated every three layers of the atom layers of graphite.

Further, since X-rays having a wavelength of 3.5 nm or less can transmit through air, detecting devices with high performance for medical use or industrial use, with small size and light weight can be realized while applying features of coherent X-rays.

Further, since coherent X-rays having a wavelength of 1 nm or less can be obtained by this invention, there can be constructed a steric laser X-ray CT device, X-ray steric measuring device, etc., which may give remarkably great effects socially and economically.

This invention is illustrated by way of the following Examples.

Example 1

An intercalation compound was produced by using apparatuses shown in Figs. 1, and 2. A graphite crystal 1 of single crystal of 20 x 12 x 1 mm in size (obtained by pyrolysis of a hydrocarbon and annealed for a long time at high temperatures while pressed at the C-axis direction) was placed in chamber A of glass chamber 10 made of silica glass and 2 ml of $SiCl_4$ (2) (purity 99.999%) was poured into chamber B. The air in the glass chamber 10 was removed from a glass tube C, which was sealed by melting the glass tube. The glass chamber 10 was inserted into an electric furnace 3 as shown in Fig. 2. The chamber A containing the single crystal of graphite 1 was heated at 300 °C and the chamber B containing $SiCl_4$ (2) was heated at 270 °C. After allowing to stand at this state for 10 days, the glass chamber 10 was cooled at a rate of 1 °C/min.

The X-ray diffraction method of the resulting single crystal of graphite 1 showed that a molecular layer of $SiCl_4$ was intercalated in every three carbon atomic layers.

The measurement of the Hall effect of this sample revealed that the hole density at room temperature was 3×10^{19} cm^{-3}. When the temperature was lowered from room temperature, the inversion of the positive and negative marks of the Hall coefficient took place at 170 K and the electron density at 20 K was 1×10^{19} cm^{-3}. Therefore, the measurement of temperature was possible from the Hall electron density and the positive or negative mark of Hall coefficient. In this case, the accuracy of measurement was as high as ±0.001 K.

Other halides of elements belonging to the group IV were also able to be similarly intercalated in graphite. Intercalation compounds suitable for practical use could be produced by using a heating temperature of graphite (T_G) of 150 to 400 °C, a heating temperature of the halides of 0 to 70 °C lower than the T_G, and a cooling rate of 0.1 to 10 °C/sec.

Example 2

Intercalation compounds were produced in the same manner as described in Example 1 by heating a single crystal of graphite in the chamber A of the glass chamber 10 at 100 to 400 °C (T_G) and heating $SiCl_4$ in the chamber B at a temperature 0 to 70 °C lower than T_G in the electric furnace shown in Fig. 2. Intercalation compounds having

the same properties as those obtained in Example 1 were obtained when the heating time was 40 hours or longer.

Example 3

A single crystal of graphite 1 was placed in the chamber A of the glass chamber 10 and $SiCl_4$ was poured into the chamber B shown in Fig. 1 in the same manner as described in Example 1. Then, Cl_2 gas was introduced into the glass chamber, followed by sealing of the glass tube C by heating. The same heating conditions as described in Example 2 were employed. As a result, an intercalation compound intercalating $SiCl_4$ and having the same properties as those obtained in Examples 1 and 2 was obtained.

The pressure of Cl_2 gas in the glass chamber at room temperature was preferable in the range of 5.3 to 8 Pa, but the intercalation of $SiCl_4$ was possible when the Cl_2 pressure was 2 bars or lower.

Example 4

The process of Example 1 was repeated except for using graphoil (graphite obtained by pyrolysis of a hydrocarbon, having a domain structure in the c plain and being a polymorph good in orientation at the c-axis direction) in place of a single crystal of graphite. An intercalation compound intercalating $SiCl_4$ having the same properties as those obtained in Examples 1 to 3 was obtained.

Example 5

The process of Example 1 was repeated except for using $GeCl_4$ in place of $SiCl_4$ to give an intercalation compound intercalating $GeCl_4$.

Example 6

The process of Example 1 or 3 was repeated except for using $SnCl_4$ in place of $SiCl_4$ to give an intercalation compound intercalating $SnCl_4$. In the case of repeating the process of Example 3, better results in the intercalation were obtained, when the Cl_2 gas pressure in the glass chamber was higher than that in Example 3.

The resulting intercalation compound had properties at the layer direction similar to those of metals and showed a large change in interlayer current depending on the voltage, light and pressure.

Example 7

A single crystal of graphite 1 and 3 ml of $SiCl_4$ (2) were placed in a glass chamber shown in Fig. 3. The whole temperature was adjusted by a heater 11. Then, the temperature of graphite 1 was controlled at 200 °C by using a heater 12. $SiCl_4$ (2) was vaporized by using a heater 13 and moved to the graphite 1 by passing air from an air inlet 14. Numeral 15 is a gas outlet, and numeral 16 is a burner for sealing a gas outlet. Then, the temperature of graphite 1 was raised to 300 °C in one hour and maintained for another one hour. During said 2 hours, the $SiCl_4$ (2) seemed to be intercalated between atom layers of carbon atoms of the graphite crystal 1. Then, the temperature of graphite 1 was lowered to 50 °C in 30 minutes by controlling the heaters 11, 12 and 13 and passing air, and to room temperature in 3.5 hours. The resulting intercalation compound was remarkably stable and very small in crystal defects, and showed strong anisotropy in photoconductivity.

Example 8

When an intercalation compound 101 obtained by intercalating $SiCl_4$ in a single crystal of graphite was radiated with synchroton radiation (SOR) 109 having a wavelength of 0.669 nm by using a SOR equipment 108 as shown in Fig. 7, coherent X-rays 104 having a wavelength of 0.66 nm were emitted. The angle between a line perpendicular to the atom layer of the intercalation compound 101 and the incident direction of SOR was effective in the range of 0° to 40°. When the angle is larger than 40°, there takes place Bragg reflection, which results in making the incidence of SOR into the intercalation compound difficult.

As the X-ray source, it was also possible to use plasma focus X-rays other than SOR.

Example 9

To an intercalation compound obtained by intercalating NaBr in a graphite single crystal, X-rays were radiated as shown in Fig. 5. The X-rays were obtained from an X-ray generating means using Mo as a target and had a wavelength of 0.227 nm.

Example 10

A pair of Si layers 107 were positioned in parallel to the atomic layer of an intercalation compound 101 obtained by intercalating $SiCl_4$ in a single crystal of graphite as shown in Fig. 8 or a pair of Si layers 107' were formed on surfaces of the intercalation compound in parallel to the atomic layer thereof as shown in Fig. 9.

The resulting devices were operated as optical resonators in individual cases.

When the thickness of Si layer was more than 10 atomic layers, there was a tendency to lower the ability of resonance. Further, needless to say, the Si layer can be formed on some substrates.

Example 11

A SiO_2 film 120 having approximately 1 μm in thickness was formed on side faces of an intercalation compound 101 obtained by intercalating $SiCl_4$ in a single crystal of graphite perpendicular to the atomic layers as shown in Fig. 10. When this sample was radiated with X-rays having a wavelength of 0.669 nm, there were obtained coherent X-rays having a wavelength of 0.66 nm.

Example 12

A polyimide resin film 120 was formed on the whole surfaces of an intercalation compound 101 obtained by intercalating $SiCl_4$ in a single crystal of graphite as shown in Fig. 11. When this sample was radiated with X-rays having a wavelength of 0.669 nm, there were obtained X-rays having a wavelength of 0.66 nm.

Example 13

A single crystal of graphite 121 (obtained by pyrolysis of a hydrocarbon and annealed for a long time at high temperatures while pressed in the c-axis direction) was placed in the chamber A and 200 mg of NaBr powder were placed in the chamber B of a glass chamber 130 as shown in Fig. 12. The air in the glass chamber 130 was removed from a glass tube C, which was then sealed by heating. The glass chamber 130 was placed in an electric furnace 133 as shown in Fig. 13. The single crystal of graphite 121 was heated at 1000 °C and NaBr 131 was heated at 1100 °C, and allowed to stand at this state for 20 days. Then, the glass chamber 130 was cooled to 100 °C at a rate of 2 °C/sec and to room temperature at a rate of 0.5 °C/sec to give an intercalation compound which was stable and suitable for use in a device for emitting coherent radiation.

Using the intercalation compound 101, a device for emitting coherent radiation such as coherent X-rays 104 in combination with a special X-ray generating means 135 using Cr as target as shown in Fig. 14 was constructed. In Fig. 14, numeral 136 is X-rays.

Intercalation compounds suitable for use in such a device can be produced by changing the heating temperature of graphite (T_G) in the range of 800 to 1500 °C, the heating temperature of the halide such as NaBr in the range of 0 to 200 °C lower than the T_G, and the cooling rate in the range of 0.5 to 20 °C/sec.

Claims

1. A device for emitting coherent radiation comprising an optical resonator containing an intercalation compound obtained by intercalating at least one molecular layer of a compound between the atomic layers of a layered crystalline substance at a constant interval of atomic layers, and an exciting energy supplying means for supplying exciting energy to the intercalation compound so as to emit coherent radiation, wherein the intercalation compound is intercalated by a halide of an element belonging to the group IVa of the periodic table or a sodium halide.

2. The device of claim 1, wherein the halide is one member selected from the group consisting of $SiCl_4$, $SnCl_4$, $GeCl_4$, $PbCl_4$, $GeBr_4$, NaCl and NaBr and the interval of atomic layers is one, two or three atom layers of graphite.

3. The device of claim 1 or 2, wherein the halide is intercalated in two or more molecular layers in the crystal layers of graphite.

4. The device of any of claims 1 to 3, wherein the energy supplying means radiates X-rays and the coherent radiation emitted is X-rays having a wavelength of 1 nm or less.

Patentansprüche

1. Gerät zum Emittieren kohärenter Strahlung, mit einem optischen Resonator, der eine durch Intercalieren mindestens einer Molekularschicht einer Verbindung zwischen die Atomschichten einer geschichteten kristallinen Substanz mit konstantem Intervall zwischen den Atomschichten gebildete Intercalationsverbindung enthält, sowie mit einer Einrichtung zum Beaufschlagen der Intercalationsverbindung mit Anregungsenergie zur Emission kohärenter Strahlung, wobei die Intercalationsverbindung durch ein Halogenid eines Elements der Gruppe IVa des Periodensystems oder ein Natriumhalogenid intercaliert ist.

2. Gerät nach Anspruch 1, wobei das Halogenid aus der $SiCl_4$, $SnCl_4$, $GeCl_4$, $PbCl_4$, $GeBr_4$, NaCl und NaBr umfassenden Gruppe ausgewählt ist und das Intervall der Atomschichten eine, zwei oder drei Graphit-Atomschichten beträgt.

3. Gerät nach Anspruch 1 oder 2, wobei das Halogenid in zwei oder mehrere Molekularschichten in den Graphit-Kristallschichten intercaliert ist.

4. Gerät nach einem der Ansprüche 1 bis 3, wobei die Energie-Beaufschlagungseinrichtung Röntgenstrahlen aussendet und die emittierte kohärente Strahlung Röntgenstrahlen mit einer Wellenlänge von 1 nm oder weniger ist.

Revendications

1. Dispositif servant à émettre un rayonnement cohérent, comprenant un résonateur optique contenant un composé à intercalation obtenu en intercalant au moins une couche moléculaire d'un composé entre les couches atomiques d'une substance cristalline à l'état de couches à un intervalle constant de couches atomiques, et des moyens d'alimentation en énergie d'excitation pour appliquer une énergie d'excitation au composé à intercalation de manière à obtenir l'émission d'un rayonnement cohérent, le composé à intercalation comportant, en tant que composé intercalé, un halogénure d'un élément appartenant au groupe IVa du tableau périodique ou un halogénure de sodium.

2. Dispositif selon la revendication 1, dans lequel l'halogénure est un élément choisi dans le groupe comprenant $SiCl_4$, $SnCl_4$, $GeCl_4$, $PbCl_4$, $GeBr_4$, NaCl et NaBr, et l'intervalle de couches atomiques est égal à 1, 2 ou 3 couches atomique de graphite.

3. Dispositif selon la revendication 1 ou 2, dans lequel l'halogénure est intercalé dans deux ou un plus grand nombre de couches moléculaires dans les couches cristallines de graphite.

4. Dispositif selon l'une quelconque des revendications 1 à 3, dans lequel les moyens d'alimentation en énergie émettent des rayons X, et le rayonnement cohérent émis est formé de rayons X ayant une longueur d'onde de 1 nm ou moins.

FIG. 1

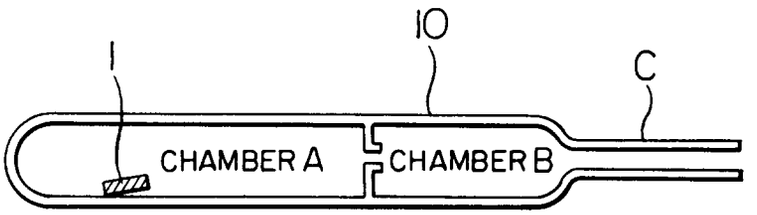


FIG. 2

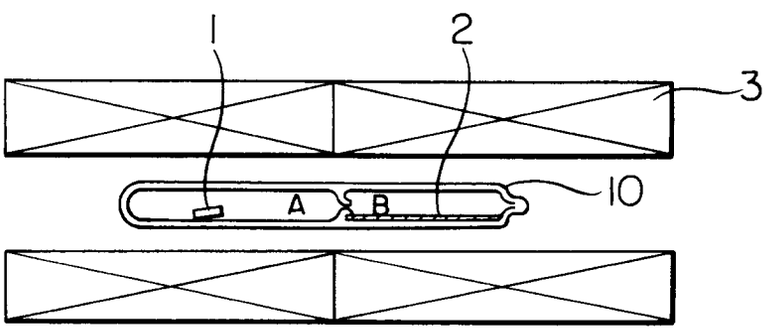


FIG. 3

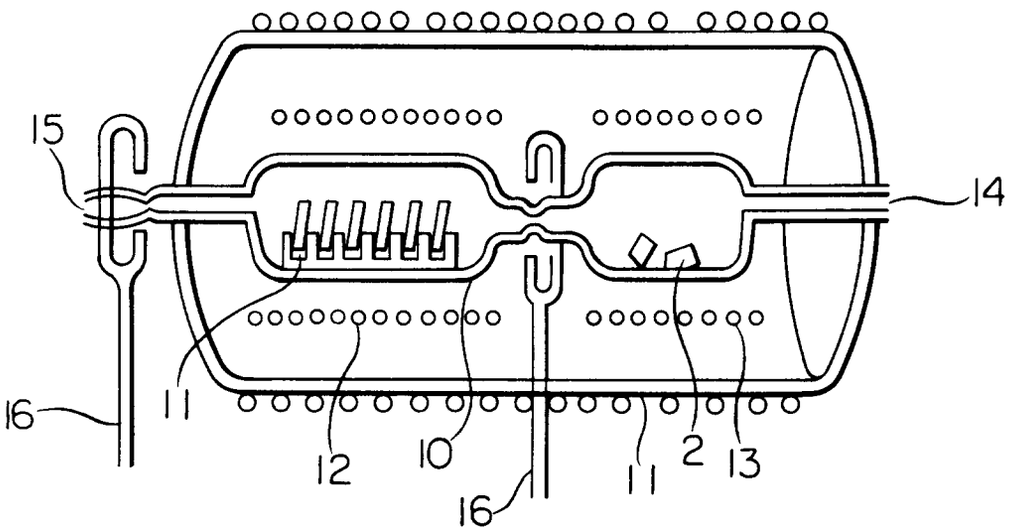

15
14
16
11
12
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16
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2
13

FIG. 4

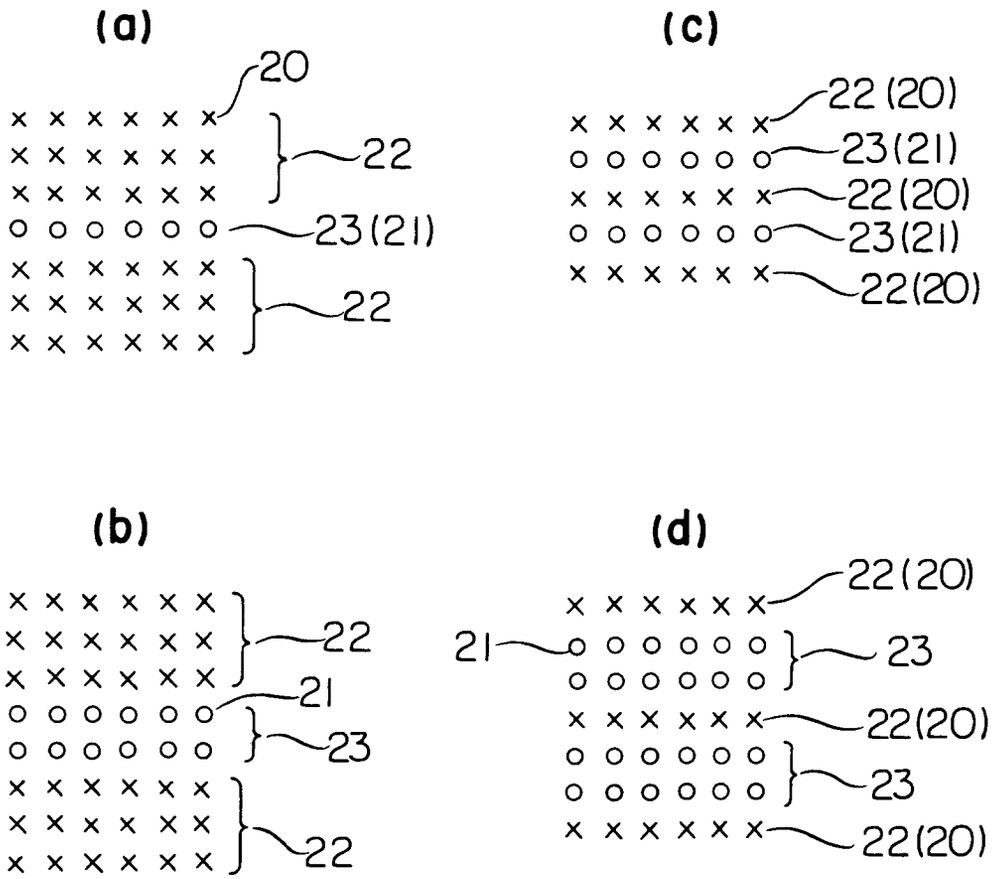

(a)
20
22
23(21)
22
(c)
22(20)
23(21)
22(20)
23(21)
22(20)
(b)
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23
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(d)
22(20)
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22(20)

FIG. 5

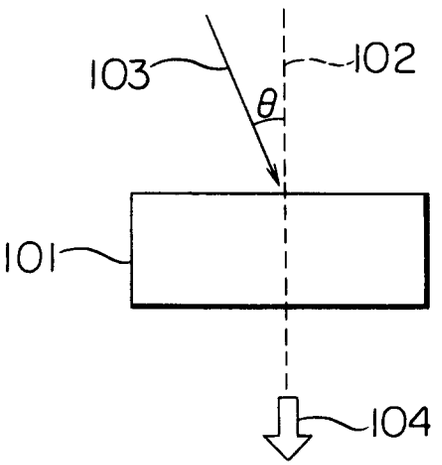


FIG. 6

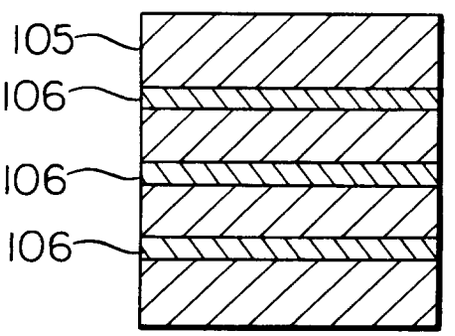


FIG. 7

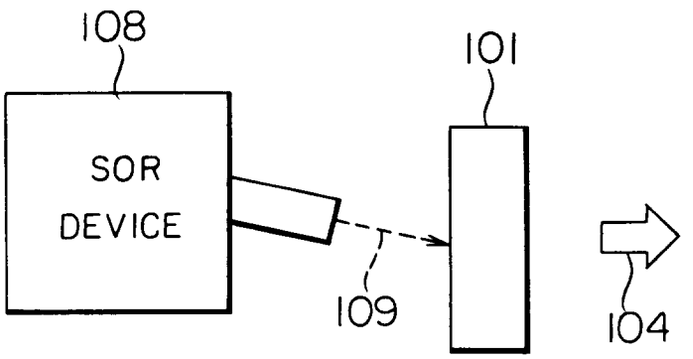


FIG. 8

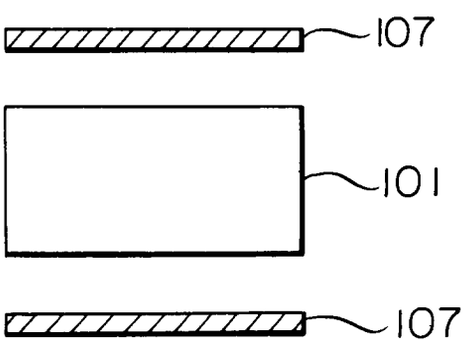


FIG. 9

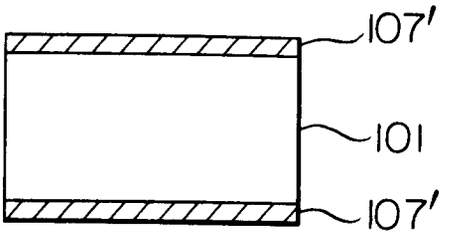


FIG. 10

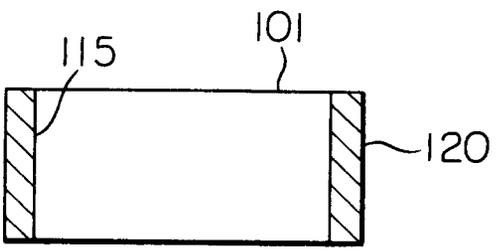


FIG. 11

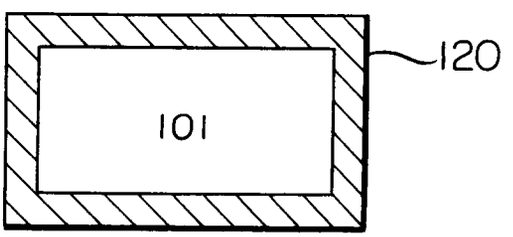


FIG. 12

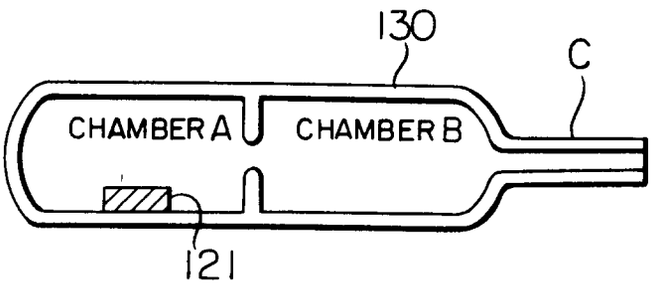


FIG. 13

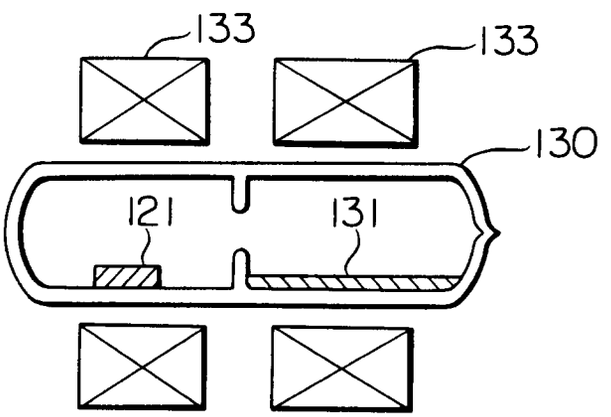


FIG. 14

